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## Analytical Report

### PFOA and PFOS Analysis of Wild Turkey Serum Samples by LC/MS/MS

**MPI Report No. L0019898**

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#### *Testing Laboratory*

MPI Research, Inc.  
3058 Research Drive  
State College, PA 16801

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#### *Requester/Project Manager*

Dena Haverland  
Dalton Utilities  
PO BOX 869  
Dalton, GA 30722  
Phone: 706-529-1010

## **1 Introduction**

Results are reported for the analysis of turkey serum samples received at MPI Research from Dalton Utilities. The MPI Research study number assigned to the project is L0019898. Table I lists the target analytes quantitated for the samples.

Table I. Target Analytes for Quantitation

Compound Name	Acronym
Perfluorooctanoic Acid	C8 Acid or PFOA
Perfluorooctanesulfonate	C8 Sulfonate or PFOS

## **2 Sample Receipt**

Two samples were received from Dena Haverland at Dalton Utilities for this study. The samples were collected on December 16, 2009. The samples arrived on December 31, 2009 via Fedex and were logged in under MPI Research login number L0019898. The shipment was received frozen on dry ice. The samples were stored frozen at approximately -80°C from receipt until analysis. Chain-of-custody information is presented in Attachment A.

## **3 Methods - Analytical and Preparatory**

### **3.1 Serum Sample Preparation**

- 3.1.1. Measure 1 mL of serum sample into a 50 mL disposable centrifuge tube and fortify, if appropriate. Add 0.2 mL of a 100 ng/mL WIS for a final concentration of 0.5 ng/mL.
- 3.1.2. Add water to sample for a final volume of 20 mL. Cap tightly and vortex for ~1 minute.
- 3.1.3. Transfer 1 mL of the sample using a disposable pipette into 15 mL disposable centrifuge tubes. Add 5 mL of ACN and shake for ~20 minutes on a wrist action shaker.
- 3.1.4. Centrifuge tubes at ~3000 rpm for ~ 5 minutes. Carefully decant supernatant into a 50 mL disposable centrifuge tube and add 35 mL of water.
- 3.1.5. Place the unconditioned SPE columns on the vacuum manifold. Condition the SPE columns by passing ~ 10 mL of methanol through the column followed by ~ 5 mL of water. The washes may be pulled through the SPE column using vacuum at a flow rate of ~1 drop/sec or may be allowed to pass through the column unaided. Discard all washes. Do not allow the column to dry.
- 3.1.6. Load the sample onto a conditioned SPE column . Discard the eluate. Any analyte residues will be trapped on the SPE column at this point.
- 3.1.7. Elute with 2 mL of methanol. Collect 2 mL of elute into a graduated 15 mL centrifuge tube.

Note: When sample dilution was required, the sample was diluted in control turkey serum prior to extraction.

### 3.2 Sample Analysis by LC/MS/MS

In High Pressure Liquid Chromatography (HPLC), an aliquot of extract is injected and passed through a liquid-phase chromatographic column. Based on the affinity of the analyte for the stationary phase in the column relative to the liquid mobile phase, the analyte is retained for a characteristic amount of time. Following HPLC separation, mass spectrometry provides a rapid and accurate means for analyzing a wide range of organic compounds. Molecules are ionized, fragmented, and detected. The ions characteristic of the compounds are observed and quantitated against external calibration standards.

An HP1100 system interfaced to an Applied Biosystems API 4000 LC/MS/MS was used to analyze the sample extracts for quantitation. A gradient elution through a Phenomenex Luna 3 $\mu$  C8(2) Mercury, 20 x 4.0 mm column was used for separation.

The following gradient was performed:

Mobile Phase (A): 2mM Ammonium Acetate in Water  
Mobile Phase (B): Methanol

Time	%A	%B
0.0	90	10
0.5	90	10
2.0	10	90
5.0	10	90
5.1	0	100
6.0	0	100
6.1	90	10
10.0	90	10

The following parameters were used for operation of the mass spectrometer:

Parameter	Setting
Ionization Mode	Electrospray
Polarity	Negative
Transitions Monitored	413→369 (PFOA) 413→219 (PFOA Confirmation) 499→80 (PFOS) 499→99 (PFOS Confirmation) 415→370 (Internal Std. $^{13}\text{C}$ PFOA (m+2)) 503→80 (Internal Std. $^{13}\text{C}$ PFOS (m+4))
Gas Temperature	450°C

## **4 Analysis by LCMSMS**

### **4.1 Calibration**

For the serum sample analysis, a 6-point calibration curve was analyzed throughout the analytical sequence for PFOA and PFOS. The calibration points were prepared at 0.1, 0.2, 0.5, 1.0, 2.0, 5.0 ng/mL (ppb) each containing 1.0 ng/mL  $^{13}\text{C}$ -PFOA ( $m+2$ ) and  $^{13}\text{C}$ -PFOS ( $m+4$ ).

The ratio of the analyte concentration to the IS concentration versus the ratio of the analyte instrument response (area) to the IS response (area) was plotted for each point. Using linear regression with  $1/x$  weighting, the slope, y-intercept and coefficient of determination ( $r^2$ ) were determined. A calibration curve is acceptable if  $r^2 \geq 0.985$ .

For the results reported here, calibration criteria were met. The calibration curves are included in the raw data in Attachment C.

### **4.2 Laboratory Control Spikes**

Laboratory control spikes in the analytical set were prepared during each extraction set by adding a known concentration of the analyte to turkey serum controls. Laboratory control spikes are used to assess method accuracy. The laboratory control spikes must show recoveries between 70-130% or the data is rejected. For the results reported here, the laboratory control spikes were within the acceptable range. Laboratory control spike recoveries are given in Attachment B.

### **4.3 Matrix Spikes**

One matrix spike was prepared by adding a known concentration of the target analyte to a sample. Matrix spikes are used to assess method accuracy in the matrix. The matrix spikes should show recoveries between 70-130%. For the results reported here, the matrix spike was within the acceptable range.

Note: Due to the endogenous level of PFOS in the sample, the sample was diluted in control turkey serum prior to the fortification of the 5000 ng/mL spike.

### **4.4 Laboratory Duplicates**

One sample was prepared in duplicate and analyzed. Duplicate results are given along with the sample results in Attachment B.

## **5 Data Summary**

Due to an interfering matrix peak at the 499 $\rightarrow$ 80 m/z transition, the 499 $\rightarrow$ 99 m/z confirmation transition was used for quantitation. The interfering matrix peak was not present at the 499 $\rightarrow$ 99 m/z confirmation transition.

Please see Attachment B for a detailed listing of the analytical results. For the serum samples the results are reported in parts per billion (ng/mL) on an as-received basis.

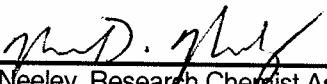
## **6 Data/Sample Retention**

Samples are disposed of 60 days after the report is issued unless otherwise specified by the project manager. All electronic data is archived on retrievable media and hard copy reports are stored in data folders maintained by MPI Research. Hardcopy data is stored for a minimum of five years. The client will be notified 30 days prior to the disposal of hardcopy data.

## **7 Attachments**

- 7.1 Attachment A: Chain of Custody
- 7.2 Attachment B: Analytical Results
- 7.3 Attachment C: Raw Analytical Data

## **8 Signatures**

  
\_\_\_\_\_  
Mark Neeley, Research Chemist Associate II

2-16-10  
Date

  
\_\_\_\_\_  
Robert Zhu, Manager, Analytical

2/17/10  
Date

**A**

## Login

**Login Group: L0019898**

Login #:	20012	Conform COC Sample:	True
Project:	P0005380	Conform COC:	True
Company Name:	Dalton Utilities	Conform Sample:	True
Submitted By:	Dena Haverland	Conform Request:	True
Login Type:	Immediate Receipt of Samples		
Started:	True		
Date Start:	12/31/2009		
Due Date:	01/10/2010		
Login Initiated:	12/31/2009		
Received By:	Kyle, Matt		
Spread Sample:			
Label:			
MPI SD/PI:	Zhu, Xiang		
Project Title/Type: PFOA AND PFOS ANALYSIS OF TURKEY BLOOD, MUSCLE AND LIVER / ROUTINE			
Login Notes:			

### Packages / Containers

Package	Carton	Date / Condition		Shipper / ID	Temp. Control/Temp.	Direction / Handled By
K0022627		Received Date: 12/31/09 11:33 Package & Contents Uncompromised		FEDEX 8694 2057 8384	Dry Ice -79.0	RECEIVED Kyle, Matt
Container #	Gross Weight	pH	Container Type	Preservative	Mfg. Lot	Mfg. ID
C0473186	4.10 g		2 ml clear plast vial	NONE		
C0473187	3.00 g		2 ml clear plast vial	NONE		
C0473188	4.60 g		2 ml clear plast vial	NONE		
C0473189	4.20 g		2 ml clear plast vial	NONE		

### Samples

Sample ID	Container	Matrix	System	System Matrix	Sample	Date Sampled	Date Due
L0019898-0001	C0473186	LIQUID	Turkey	Serum	Wild Turkey #2 4yr Male - Serum	12/16/2009	01/10/2010
	C0473187						
L0019898-0002	C0473188	LIQUID	Turkey	Serum	Wild Turkey #5 1yr Male - Serum	12/16/2009	01/10/2010
	C0473189						

Login Reviewed By:



Date/Time:

2/11/10 1519.





## Sample Submittal

MPI Research Contact: Daniel Wright

### Send Report To:

Company: Dalton Utilities  
1200 VD Parrott JR Parkway, PO Box 869  
Address:

City, State, ZIP: Dalton, GA 30722-0869

Attention: Dena Haverland

Phone #: 706-529-1010

Fax #: 706-529-1271

Email: dhaverland@dutil.com

Study/Job #:

Signature/Date:

Printed Name:

Please fax this form before sending samples.

Please send samples to shipping and receiving:  
3048 Research Drive, State College, PA 16801  
T: (814) 272-1039 • F: (814) 272-1019

### Turnaround time (TAT) requirements:

Results Due Date: 30 days

Preliminary Results Format: Verbal  Email  Fax

Report Due Date: 30 days

### Storage Conditions

Room temperature  
Refrigerator  
 Freezer  
Ultra Low freezer  
Desiccated  
Lighting required

Stability (°C/%RH):

Stability time period:

### Safety Information

Special handling:

MSDS attached

Controlled substance:

HAZARDS:

Please fill in the diamond HMIS/NFPA  
(0-4) if appropriate



Client ID# Description	Lot/ Control #	Amt. Sent/ Weight	# of Bottles	Matrix	Date & Time	Tests Requested
1 Wild Turkey #2 4yr Male - Serum		3ml	2	Turkey	12/16/09 11:15 am	PFOA/PFOS
2 Wild Turkey #2 4yr Male - Muscle		408gm	1 bag	Turkey	12/16/09 11:15 am	PFOA/PFOS
3 Wild Turkey #2 4yr Male - Liver		131gm	1 bag	Turkey	12/16/09 11:15 am	PFOA/PFOS
4 Wild Turkey #5 1yr Male - Serum		4ml	2	Turkey	12/16/09 12:00 pm	PFOA/PFOS
5 Wild Turkey #5 1yr male - Muscle		442gm	1 bag	Turkey	12/16/09 12:00 pm	PFOA/PFOS
6 Wild Turkey #5 1yr Male - Liver		82gm	1 bag	Turkey	12/16/09 12:00 pm	PFOA/PFOS
7						
8						
9						
10						

PO #: \_\_\_\_\_

Notes:

Relinquished by	Date	Time	Received by	Date	Time
Daniel Wright	12/21/09	3:00pm	Wyl	12/21/09	1:33

V0002836 2





## TEMPORARY SAMPLE STORAGE FORM

To be completed during ExyLIMS Login

Project #: PS195

Login #: 19898

Initials / Date: MJK 12/31/09

One form to be completed for each package

Date / Time Received: 12/22/09 1133

Received By: M. Kuhn

Shipper: Fed Ex

Shipper Package ID: 8694 2057 8384

Temperature (deg C) / Thermometer ID: -79.0° IN0000448

Temperature Control Method: Dry Ice

Temporary Storage Location: Freezer 32

Condition of sample(s):

- Good – Package and contents uncompromised
- Fair – Package damaged / contents uncompromised
- Poor – Package and contents compromised

Notes:

**B**



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## Analytical Report

### Summary of Fluorochemical Residues in Serum Samples

Sample ID	PFOA Perfluorooctanoic Acid	PFOS Perfluorooctanesulfonate
	Analyte Found (ng/mL, ppb)	Analyte Found (ng/mL, ppb)
Wild Turkey # 5 1yr male-serum	33.1	2820
Wild Turkey # 5 1yr male-serum*	33.5	2570
Wild Turkey # 2 4yr male-serum	18.5	2090

\*Laboratory Duplicate

ND = Not detected = Response is below the LOD of 1.0 ng/mL (ppb).

NQ = Not quantifiable = Response is between the LOD and the LOQ of 10 ng/mL (ppb).



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### Recovery Summary of Fluorochemical Residues in Serum Samples

Sample Description	Amount Spiked (ng/mL)	PFOA			PFOS		
		Amt Found in Sample (ng/mL)	Amount Recovered (ng/mL)	Recovery (%)	Amt Found In Sample (ng/mL)	Amount Recovered (ng/mL)	Recovery (%)
LCS A (Data set 020510A) 10 ng/mL	10	ND	11.1	111	ND	12.1	121
LCS B (Data set 020510A) 50 ng/mL	50	ND	52.1	104	ND	50.4	101
LCS A (Data set 020810A) 5000 ng/mL	5000	N/A	N/A	N/A	ND	6386	128
LCS B (Data set 020810A) 5000 ng/mL	5000	N/A	N/A	N/A	ND	5984	120
Wild Turkey # 5 1yr male-serum (L19898-2 Spk C, 50 ng/mL Lab Spike)	50	33.1	87.5	109	2820	**	**
Wild Turkey # 5 1yr male-serum (L19898-2 Spk C, 5000 ng/mL Lab Spike)	5000	N/A	N/A	N/A	2820	7770	99

ND = Not detected = Response is below the LOD of 1.0 ng/mL.

NQ = Not quantifiable = Response is between the LOD and the LOQ of 10 ng/mL.

\*\* The endogenous level of PFOS in the sample significantly exceeds the spiking level, therefore an accurate recovery cannot be calculated.

**C**

**RAW DATA REPORT**

Sponsor Study No:	NA	Limit of Quantitation:	10 ng/mL	Set No:	020510A
MPI Study No:	L19898	Injection Volume:	15 µL	Analyst:	Mark Neetley
Analyte:	PFOA	Matrix:	Wild Turkey Serum	Instrument Type:	LC/MS/MS Unit # 8
Ions Monitored:	413 -> 369			Extraction Date:	02/05/10
Site:	NA			Analyzed on:	02/05/10

MPI Research ID	Sponsor ID	Sample Code	Sample Index No.	Internal				Internal		Amount		
				Std. Conc. (ng/mL)	Std. Conc. (ng/mL)	Aliquot Factor (AF)	Dilution Factor (DF)	Peak Area	Standard Peak Area	Analyte Found (ng/mL)	Analyte Added (ng/mL)	Recovery (%)
SS33618	-	CS	1	0.100	1.0	-	-	53017	607243	-	-	-
SS33617	-	CS	2	0.200	1.0	-	-	96383	563452	-	-	-
SS33616	-	CS	3	0.500	1.0	-	-	223938	551155	-	-	-
SS33615	-	CS	4	1.00	1.0	-	-	441742	527638	-	-	-
SS33614	-	CS	5	2.00	1.0	-	-	879289	527427	-	-	-
SS33613	-	CS	6	5.00	1.0	-	-	1901673	497318	-	-	-
Methanol Wash	-	W	7	-	-	-	-	2117	0	-	-	-
Methanol Wash	-	W	8	-	-	-	-	0	0	-	-	-
Control	MC4650 Wild Turkey Serum Control	C	9	-	0.5	40	1	7066	317343	ND	-	-
LCS A	MC4650 Wild Turkey Spike A, 10 ng/mL	LCS	10	-	0.5	40	1	143178	319191	11.1	10	111
LCS B	MC4650 Wild Turkey Spike B, 50 ng/mL	LCS	11	-	0.5	40	1	547135	265812	52.1	50	104
L19898-2 Spk C	Wild Turkey # 5 1yr male-serum Spike C, 50 ng/ml	LF	12	-	0.5	40	1	888113	257637	87.5	50	109
L19898-2	Wild Turkey # 5 1yr male-serum	S	13	-	0.5	40	1	367001	279081	33.1	-	-
L19898-2 Dup	Wild Turkey # 5 1yr male-serum Duplicate	S	14	-	0.5	40	1	315050	237129	33.5	-	-
L19898-1	Wild Turkey # 2 4yr male-serum	S	15	-	0.5	40	1	173424	234623	18.5	-	-
SS33615	-	CCV	16	1.00	1.0	-	-	337694	406265	1.04	1.0	104

Analyte Found (ng/mL) = (((analyte peak area/IS peak area) - intercept) / slope) x IS conc.) x AF x DF

Standard Curve: Linear (1/x weighted)

$$\text{Recovery (\%)} = \frac{[\text{Analyte found (ng/mL)} - \text{Analyte found in control (ng/mL)}]}{\text{amount Analyte added (ng/mL)}} \times 100$$

Intercept = 0.0143

Slope = 0.785

Coef. Of Det. = 0.9982

CS = Calibration standard

LF = Lab fortified sample

W = Methanol Wash

CCV = Continuing Calibration Verification

FF = Field fortified sample

ND = Not detected = Response between 0 and LOD

C = Control sample

LCS = Laboratory Control Spike

NQ = Not quantifiable = Response between LOD and LOQ

S = Sample

Spreadsheet prepared by: MD / 2-10-10

**RAW DATA REPORT**

Sponsor Study No: NA  
 MPI Study No: L19898  
 Analyte: PFOS  
 Ions Monitored: 499 > 80  
 Site: NA

Limit of Quantitation: 10 ng/mL  
 Injection Volume: 15 µL  
 Matrix: Wild Turkey Serum

Set No: 020510A  
 Analyst: Mark Neeley  
 Instrument Type: LC/MS/MS Unit # 8  
 Extraction Date: 02/05/10  
 Analyzed on: 02/05/10

MPI Research ID	Sponsor ID	Sample Code	Sample Index No.	Internal				Internal		Amount	
				Std. Conc. (ng/mL)	Std. Conc. (ng/mL)	Extraction Factor	Dilution Factor	Peak Area	Peak Area	Analyte Found (ng/mL)	Analyte Added (ng/mL)
SS33618	-	CS	1	0.100	1	-	-	16834	158850	-	-
SS33617	-	CS	2	0.200	1	-	-	32666	150672	-	-
SS33616	-	CS	3	0.500	1	-	-	75047	148798	-	-
SS33615	-	CS	4	1.00	1	-	-	144667	147467	-	-
SS33614	-	CS	5	2.00	1	-	-	292035	150146	-	-
SS33613	-	CS	6	5.00	1	-	-	687643	134977	-	-
Methanol Wash	-	W	7	-	-	-	-	38	0	-	-
Methanol Wash	-	W	8	-	-	-	-	68	0	-	-
Control	MC4650 Wild Turkey Serum Control	C	9	-	0.5	40	1	38814	67513	-	-
LCS A	MC4650 Wild Turkey Spike A, 10 ng/mL	LCS	10	-	0.5	40	1	74658	65664	-	10
LCS B	MC4650 Wild Turkey Spike B, 50 ng/mL	LCS	11	-	0.5	40	1	170301	54433	-	50
L19898-2 Spk C	Wild Turkey # 5 1yr male-serum Spike C, 50 ng/mL	LF	12	-	0.5	40	1	4589621	36832	-	50
L19898-2	Wild Turkey # 5 1yr male-serum	S	13	-	0.5	40	1	4675252	38111	-	-
L19898-2 Dup	Wild Turkey # 5 1yr male-serum Duplicate	S	14	-	0.5	40	1	4144447	34008	-	-
L19898-1	Wild Turkey # 2 4yr male-serum	S	15	-	0.5	40	1	3001658	31232	-	-
SS33615		CCV	16	1.00	1	-	-	117157	108299	-	1

Analyte Found (ppb) = (((analyte peak area/IS peak area) - Intercept) / Slope) x IS conc.) x DF

Standard Curve : Linear (1/x weighted)

Recovery (%) =  $\frac{[\text{Analyte found (ng/mL)} - \text{Analyte found in control (ng/mL)}]}{\text{amount Analyte added (ng/mL)}} \times 100$

Intercept = 0.00632

Slope = 1

Coef. Of Det. = 0.999

CS = Calibration standard  
 CCV = Continuing Calibration Verification  
 C = Control sample  
 S = Sample

LF = Lab fortified sample  
 FF = Field fortified sample  
 LCS = Laboratory Control Spike

W = Methanol Wash  
 ND = Not detected = Response between 0 and LOD  
 NQ = Not quantifiable = Response between LOD and LOQ

Note: Due to an interfering peak in the 499->80 m/z transition for PFOS, the 499->99 m/z confirmation transition was used for quantitation.

Spreadsheet prepared by: Mark 12-10-10

**RAW DATA REPORT**

Sponsor Study No:	NA	Limit of Quantitation:	10 ng/mL	Set No:	020510A
MPI Study No:	L19898	Injection Volume:	15 µL	Analyst:	Mark Neely
Analyte:	PFOA Confirmation	Matrix:	Wild Turkey Serum	Instrument Type:	LC/MS/MS Unit # 8
Ions Monitored:	413 -> 219			Extraction Date:	02/05/10
Site:	NA			Analyzed on:	02/05/10

MPI Research ID	Sponsor ID	Sample Code	Sample Index No.	Internal				Internal		Amount		
				Std. Conc. (ng/mL)	Std. Conc. (ng/mL)	Extraction Factor	Dilution Factor	Peak Area	Standard Peak Area	Analyte Found (ng/mL)	Added (ng/mL)	Recovery (%)
SS33618	-	CS	1	0.100	1	-	-	6183	607243	-	-	-
SS33617	-	CS	2	0.200	1	-	-	11162	563452	-	-	-
SS33616	-	CS	3	0.500	1	-	-	26151	551155	-	-	-
SS33615	-	CS	4	1.00	1	-	-	49275	527638	-	-	-
SS33614	-	CS	5	2.00	1	-	-	100927	527427	-	-	-
SS33613	-	CS	6	5.00	1	-	-	221020	497318	-	-	-
Methanol Wash	-	W	7	-	-	-	-	0	0	-	-	-
Methanol Wash	-	W	8	-	-	-	-	0	0	-	-	-
Control	MC4650 Wild Turkey Serum Control	C	9	-	0.5	40	1	1326	317343	ND	-	-
LCS A	MC4650 Wild Turkey Spike A, 10 ng/mL	LCS	10	-	0.5	40	1	16015	319191	10.7	10	107
LCS B	MC4650 Wild Turkey Spike B, 50 ng/mL	LCS	11	-	0.5	40	1	60910	265812	50.2	50	100
L19898-2 Spk C	Wild Turkey # 5 1yr male-serum Spike C, 50 ng/mL	LF	12	-	0.5	40	1	98087	257637	83.7	50	103
L19898-2	Wild Turkey # 5 1yr male-serum	S	13	-	0.5	40	1	41292	279081	32.3	-	-
L19898-2 Dup	Wild Turkey # 5 1yr male-serum Duplicate	S	14	-	0.5	40	1	41407	237129	38.2	-	-
L19898-1	Wild Turkey # 2 4yr male-serum	S	15	-	0.5	40	1	21842	234623	20.2	-	-
SS33615		CCV	16	1.00	1	-	-	38765	406265	1.04	1.0	104

Analyte Found (ppb) = (((analyte peak area/IS peak area) - intercept) / slope) x IS conc.) x DF

Recovery (%) =  $\frac{[\text{Analyte found (ng/mL)} - \text{Analyte found in control (ng/mL)}]}{\text{amount Analyte added (ng/mL)}} \times 100$

Standard Curve : Linear (1/x weighted)

Intercept = 0.00163

Slope = 0.09

Coef. Of Det. = 0.9990

CS = Calibration standard

LF = Lab fortified sample

W = Methanol Wash

CCV = Continuing Calibration Verification

FF = Field fortified sample

ND = Not detected = Response between 0 and LOD

C = Control sample

LCS = Laboratory Control Spike

NQ = Not quantifiable = Response between LOD and LOQ

Note: The results for the 413->219 m/z transition are reported for confirmation of the 413->369 m/z transition.

Spreadsheet prepared by: MDL 1 Z-10-A

**RAW DATA REPORT**

Sponsor Study No:	NA	Limit of Quantitation:	10 ng/mL	Set No:	020510A
MPI Study No:	L19898	Injection Volume:	15 µL	Analyst:	Mark Neely
Analyte:	<b>PFOS Confirmation</b>	Matrix:	Wild Turkey Serum	Instrument Type:	LC/MS/MS Unit # 8
Ions Monitored:	499 -> 99			Extraction Date:	02/05/10
Site:	NA			Analyzed on:	02/05/10

MPI Research ID	Sponsor ID	Sample Code	Sample No.	Internal				Internal Standard Peak Area	Analyte Found (ng/mL)	Amount Added (ng/mL)	Amount Recovery (%)
				Std. Conc. (ng/mL)	Std. Conc. (ng/mL)	Extraction Factor	Dilution Factor				
SS33618	-	CS	1	0.100	1	-	-	2887	158850	-	-
SS33617	-	CS	2	0.200	1	-	-	4463	150672	-	-
SS33616	-	CS	3	0.500	1	-	-	11622	148798	-	-
SS33615	-	CS	4	1.00	1	-	-	22507	147467	-	-
SS33614	-	CS	5	2.00	1	-	-	43222	150146	-	-
SS33613	-	CS	6	5.00	1	-	-	101140	134977	-	-
Methanol Wash	-	W	7	-	-	-	-	0	0	-	-
Methanol Wash	-	W	8	-	-	-	-	57	0	-	-
Control	MC4650 Wild Turkey Serum Control	C	9	-	0.5	40	1	974	67513	ND	-
LCS A	MC4650 Wild Turkey Spike A, 10 ng/mL	LCS	10	-	0.5	40	1	6016	65664	12.1	10
LCS B	MC4650 Wild Turkey Spike B, 50 ng/mL	LCS	11	-	0.5	40	1	20431	54433	50.4	50
L19898-2 Spk C	Wild Turkey # 5 1yr male-serum Spike C, 50 ng/mL	LF	12	-	0.5	40	1	719324	36832	**	50
L19898-2	Wild Turkey # 5 1yr male-serum	S	13	-	0.5	40	1	718801	38111	*	-
L19898-2 Dup	Wild Turkey # 5 1yr male-serum Duplicate	S	14	-	0.5	40	1	635614	34008	*	-
L19898-1	Wild Turkey # 2 4yr male-serum	S	15	-	0.5	40	1	438055	31232	*	-
SS33615	-	CCV	16	1.00	1	-	-	17030	108299	1.05	1.0

Analyte Found (ppb) = (((analyte peak area/IS peak area) - intercept) / slope) x IS conc. x DF

Standard Curve : Linear (1/x weighted)

$$\text{Recovery (\%)} = \frac{[\text{Analyte found (ng/mL)} - \text{Analyte found in control (ng/mL)}]}{\text{amount Analyte added (ng/mL)}} \times 100$$

Intercept = 0.00242

Slope = 0.148

Coef. Of Det. = 0.9992

CS = Calibration standard

LF = Lab fortified sample

W = Methanol Wash

CCV = Continuing Calibration Verification

FF = Field fortified sample

ND = Not detected = Response between 0 and LOD

C = Control sample

LCS = Laboratory Control Spike

NQ = Not quantifiable = Response between LOD and LOQ

Note: The results for the 499->99 m/z transition are used for quantitation due to an interfering peak in the 499->80 m/z transition.

\* Sample response exceeds the calibration range, dilution required, see set no 020810A for analysis

\*\* The endogenous level of PFOS in the sample significantly exceeds the spiking level, therefore an accurate recovery cannot be calculated. The sample was fortified at a higher level and was re-extracted and re-analyzed, see set no. 020810A for analysis.

Spreadsheet prepared by: Mark Neely



3058 Research Drive  
State College, PA 16801

Phone: 814-272-1039  
Fax: 814-231-1580

### Internal Chain of Custody/Fortification Sheet

MPI Research Study Number:

L19898

Matrix: Wild Turkey Serum

Sponsor Study/Protocol No.:

NA

The samples listed below were removed from Freezer No. 11

Time 8:40 AM

Date 2-5-10

Initials MWD

CLIENT SAMPLE ID	MPI RESEARCH ID NUMBER	VOLUME (mL)	FORTIFICATION (ng)
na	MC4650 Control	1.0	-
na	MC4650 LCS A	1.0	10
na	MC4650 LCS B	1.0	50
Wild Turkey #5 1yr male-serum	L19898-2 Spk C	1.0	50
Wild Turkey #5 1yr male-serum	L19898-2	1.0	-
Wild Turkey #5 1yr male-serum	L19898-2 Dup	1.0	-
Wild Turkey #2 4yr male-serum	L19898-1	1.0	-
-	-	-	-
-	-	-	-
-	-	-	-
-	-	-	-
-	-	-	-
-	-	-	-
-	-	-	-
-	-	-	-
-	-	-	-
-	-	-	-
-	-	-	-
-	-	-	-
-	-	-	-
-	-	-	-
-	-	-	-
-	-	-	-
-	-	-	-
-	-	-	-

	Spiking Solution Used	Volume Used for Spiking	Initial/Date
MC4650 LCS A	SS0033612 (100 ng/mL)	100 µL (50-250µL auto-pipette)	<u>MWD</u> /2-5-10
MC4650 LCS B	SS0033611 (1000 ng/mL)	50 µL (50-250µL auto-pipette)	<u>MWD</u> /2-5-10
L19898-2 Spk C	SS0033611 (1000 ng/mL)	50 µL (50-250µL auto-pipette)	<u>MWD</u> /2-5-10
All Samples	SS0032894 (100 ng/mL)	200 µL (50-250µL auto-pipette)	<u>MWD</u> /2-5-10

All samples were measured using a digital autopipet IN 14K.

Time 12:30 PM

Date 2-5-10

Initials MWD

After measuring samples were returned to Freezer No. 40

Time 1:05 PM

Date 2-5-10

Initials MWD

#### Comments:

#### Analysis Summary:

Data Set: 020510A  
Data Set: -  
Data Set: -

Initials/Date: MWD / 2-5-10  
Initials/Date: - / -  
Initials/Date: - / -

Set extraction/analysis data verified by: BB

Date: 2/16/10  
July 02, 2007/2



3058 Research Drive  
State College, PA 16801

Phone: 814-272-1039  
Fax: 814-231-1580

MPI STUDY NUMBER: L19898  
MATRIX: Wild Turkey Serum

## SAMPLE EXTRACTION AND ANALYSIS TRACKING SHEET

METHOD: V5821

ANALYTES: PFOS & PFOA

PROTOCOL NUMBER: NA

Client ID	MPI Research ID	STEP 1	STEP 2	STEP 3	STEP 4	STEP 5	STEP 6	STEP 7	STEP 8	Reagents/ Materials	ExyLims ID
na	MC4650 Control									Acetonitrile	2-5-783
na	MC4650 LCS A									Water	2-5-785
na	MC4650 LCS B									Methanol	2-5-787
Wild Turkey #5 1yr male-serum	L19898-2 Spk C									SPE Column	2-5-794
Wild Turkey #5 1yr male-serum	L19898-2									Initials/Date	2-5-24-10
Wild Turkey #5 1yr male-serum	L19898-2 Dup										
Wild Turkey #2 4yr male-serum	L19898-1										
-	-	-	-	-	-	-	-	-	-		
-	-	-	-	-	-	-	-	-	-		
-	-	-	-	-	-	-	-	-	-		
-	-	-	-	-	-	-	-	-	-		
-	-	-	-	-	-	-	-	-	-		
-	-	-	-	-	-	-	-	-	-		
-	-	-	-	-	-	-	-	-	-		
-	-	-	-	-	-	-	-	-	-		
-	-	-	-	-	-	-	-	-	-		
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-	-	-	-	-	-	-	-	-	-		
-	-	-	-	-	-	-	-	-	-		
*Initials/Date		2-5-12	2-5-12	2-5-12	2-5-12	2-5-12	2-5-12	2-5-12	2-5-12		

STEP 1: Measure 1 mL of serum into a 50 mL disposable centrifuge tube and fortify if appropriate. Add 0.2 mL of internal standard (100 ng/mL).

STEP 2: Add water to the sample for a final volume of 20 mL. Cap tightly and vortex for ~1 minute.

STEP 3: Transfer 1.0 mL of the sample to a 15 mL centrifuge tube. Add 5 mL of ACN and shake for ~20 minutes on a wrist action shaker.

STEP 4: Centrifuge tubes at ~3000 for ~5 minutes. Carefully decant supernatant into a 50 mL disposable centrifuge tube and add 35 mL of water.

STEP 5: SPE Cleanup: Condition column with ~5 mL of methanol followed by ~5 mL of water at a rate of ~1 drop/second. Discard all washes. Do not allow column to go dry.

STEP 6: Load the sample onto the SPE Column. Discard the eluate. Elute with 2 mL of methanol into a 1.5 mL centrifuge tube. Transfer the extract into autosampler vials.

STEP 7: LC/MS/MS analysis.

STEP 8: LC/MS/MS reanalysis.

\*Initials and date under each step indicates the personnel that performed this step.

COMMENTS:

Final extracts stored in refrigerator 34 Initials: MPL Date: 2-5-12

Oct 30, 2009/1

MDR 2-5-10

Project: \\sc1wp5556\mdrive\PE SCIEX DATA\Projects\P5195 Batch:08\_020510A Tab:Sample Set:SET1 AcqMethod:P5195 10  
Sample

	Sample Name	Sample ID	Vial Position	Data File
1	SS33618	Calibration Standard, 0.1 ng/mL	1	08_020510A\020510A
2	SS33617	Calibration Standard, 0.2 ng/mL	2	08_020510A\020510A
3	SS33616	Calibration Standard, 0.5 ng/mL	3	08_020510A\020510A
4	SS33615	Calibration Standard, 1.0 ng/mL	4	08_020510A\020510A
5	SS33614	Calibration Standard, 2.0 ng/mL	5	08_020510A\020510A
6	SS33613	Calibration Standard, 5.0 ng/mL	6	08_020510A\020510A
7	Methanol Wash	Methanol Wash	91	08_020510A\020510A
8	Methanol Wash	Methanol Wash	91	08_020510A\020510A
9	Control	MC4650 Wild Turkey Serum Control	11	08_020510A\020510A
10	LCS A	MC4650 Wild Turkey Spike A, 10 ng/mL	12	08_020510A\020510A
11	LCS B	MC4650 Wild Turkey Spike B, 50 ng/mL	13	08_020510A\020510A
12	L19898-2 Spk C	Wild Turkey # 5 1yr male-serum Spike C, 50 ng/mL	14	08_020510A\020510A
13	L19898-2	Wild Turkey # 5 1yr male-serum	15	08_020510A\020510A
14	L19898-2 Dup	Wild Turkey # 5 1yr male-serum Duplicate	16	08_020510A\020510A
15	L19898-1	Wild Turkey # 2 4yr female-serum	17	08_020510A\020510A
16	SS33615	CCV, 1.0 ng/mL <i>Mtz</i>	4	08_020510A\020510A <i>(2) MDR 2-5-10</i>

# LC/MS/MS SYSTEM AND OPERATING CONDITIONS

Protocol No: NA

MPI Study No: L19898

**Instrument:** AB API 4000 Biomolecular Mass Analyzer, (LC/MS/MS #8)  
SCIEX Turbo Ion Spray Liquid Introduction Interface  
Turbo Ion spray temperature = 450 °C

**Computer:** Dell OptiPlex GX 110

**Software:** PE Sciex Analyst 1.4

**HPLC Equipment:** Hewlett Packard (HP) Series 1100  
HP Quat Pump                    HP Vacuum Degasser  
HP Autosampler                HP Column Oven

**HPLC Column:** Phenomenex Luna C8 (2) Mercury, 2cm x 4mm, 3 µm (ExyLIMS ID: MA0052622)

**Column Temperature:** 35°C

**Mobile Phase (A):** 2 mM Ammonium Acetate in Water (ExyLIMS ID: SL0047156)

**Mobile Phase (B):** Methanol (ExyLIMS ID: RE0048705)

**Injected Volume:** 15 µL

Time (min)	% A	% B	Flow Rate (µL/min)
0.0	90	10	750
0.5	90	10	750
2.0	10	90	750
5.0	10	90	750
5.1	0	100	750
6.0	0	100	750
6.1	90	10	750
10.0	90	10	750

Ions monitored:

Analyte	Parent ion	Daughter ion(s)	Dwell (secs)
PFOA	413	369	0.100
PFOS	499	80	0.100
PFOA Confirmation	413	219	0.100
PFOS Confirmation	499	99	0.100
<sup>13</sup> C PFOA (m+2)	415	370	0.100
Internal Standard			
<sup>13</sup> C PFOS (m+4)	503	80	0.100
Internal Standard			

**Analyst:** Mark Neeley      *MDN 2-5-10*  
MPI Research, Inc.  
3058 Research Drive, State College, PA 16801  
Phone: (814) 272-1039 FAX: (814) 231-1580

All Handwritten Peak ID's by: *MDN 2-10-10*

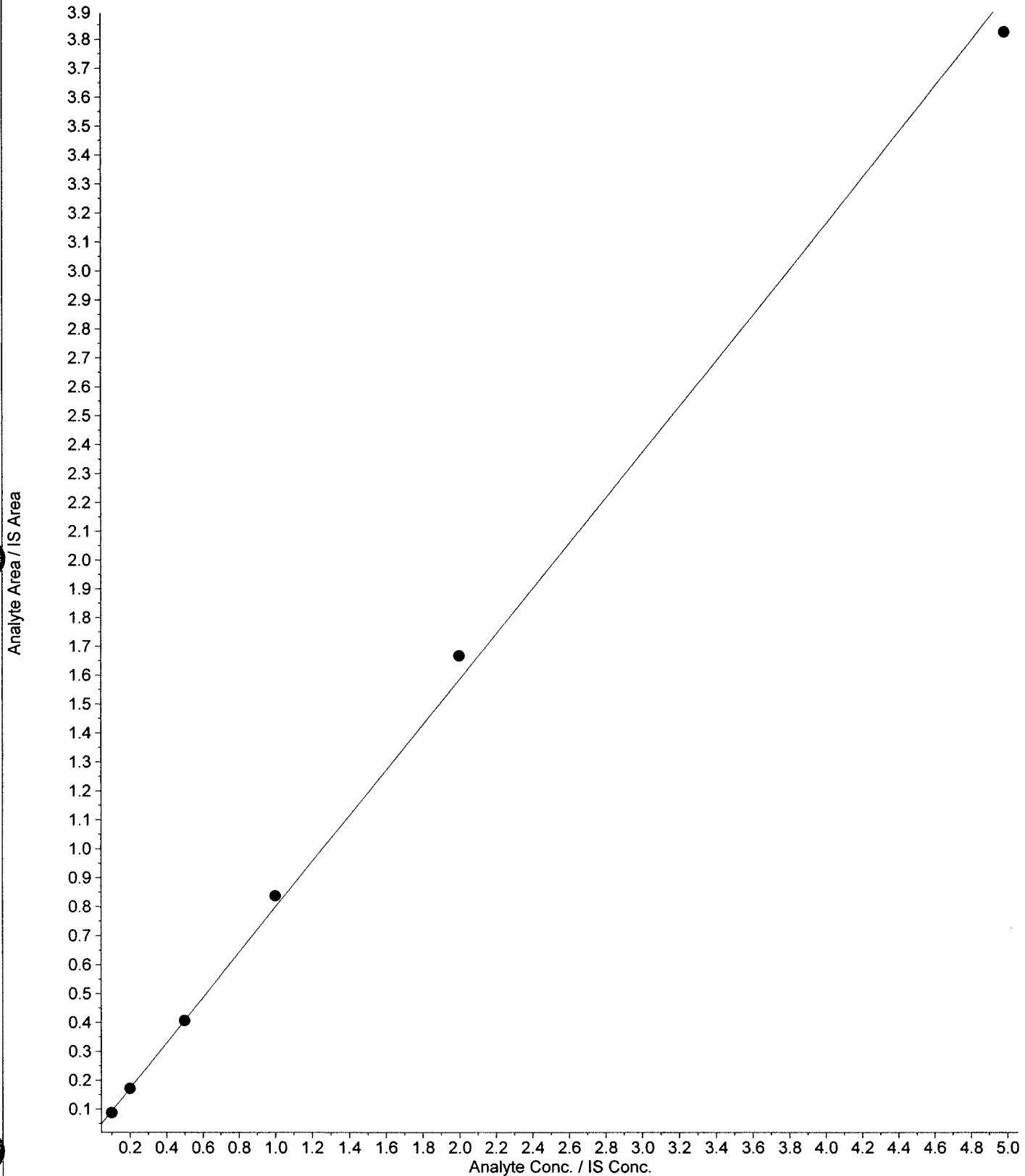
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Printing Date: Wed, Feb 10, 2010  
Analyst Version: 1.4.2

MPI Study No.: L19898  
Set No.: 020510A

Operator: Mark Neeley  
Instrument No.: LC/MS/MS # 8

MDN 2-10-10

■ 08\_020510A.rdb (PFOA): "Linear" Regression ("1 / x" weighting):  $y = 0.785 x + 0.0143$  ( $r = 0.9991$ )



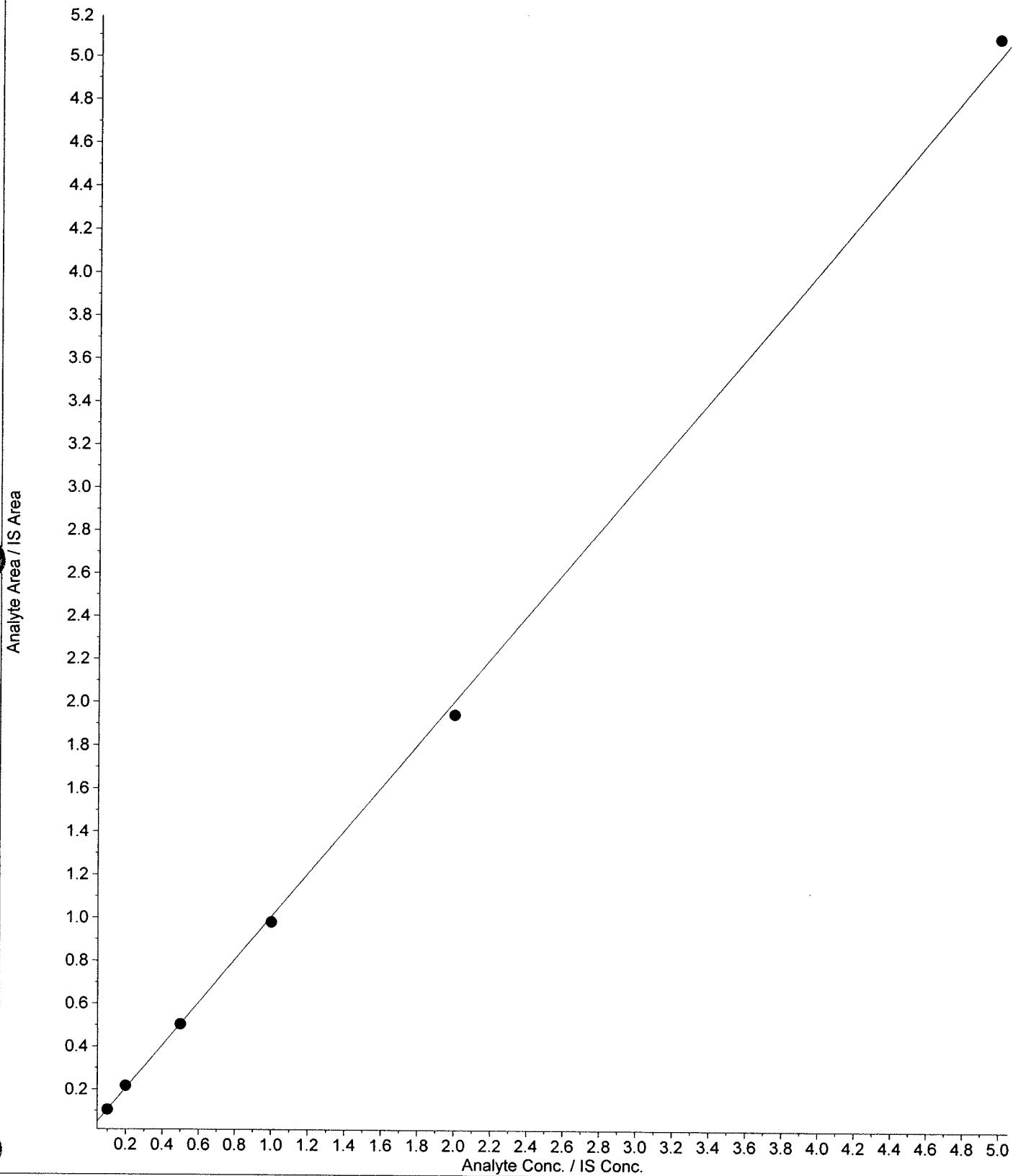
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Printing Date: Wed, Feb 10, 2010  
Analyst Version: 1.4.2

MPI Study No.: L19898  
Set No.: 020510A

Operator: Mark Neeley  
Instrument No.: LC/MS/MS # 8

MON 2-10-10

■ 08\_020510A.rdb (PFOS): "Linear" Regression ("1 / x" weighting):  $y = 1 x + 0.00632$  ( $r = 0.9997$ )



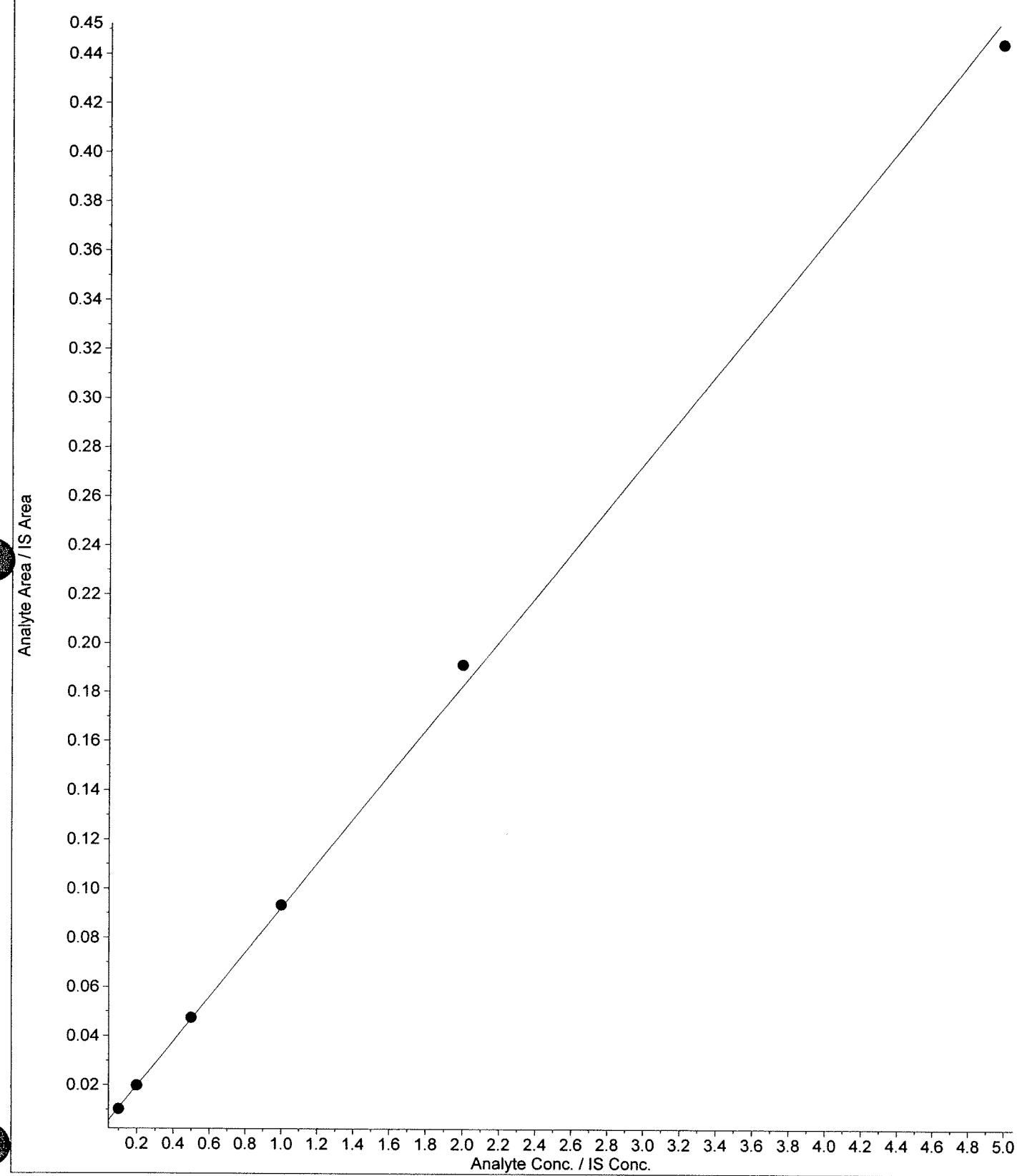
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Analyst Version: 1.4.2

MPI Study No.: L19898  
Set No.: 020510A

Operator: Mark Neeley  
Instrument No.: LC/MS/MS # 8

MON 2-10-10

■ 08\_020510A.rdb (PFOAconf): "Linear" Regression ("1 / x" weighting):  $y = 0.0906 x + 0.00163$  ( $r = 0.9995$ )



Printing Time: 8:56:51 AM  
Printing Date: Wed, Feb 10, 2010  
Analyst Version: 1.4.2

MPI Study No.: L19898  
Set No.: 020510A

Operator: Mark Neeley  
Instrument No.: LC/MS/MS # 8

MRJ 2/10/10

■ 08\_020510A.rdb (PFOSconf): "Linear" Regression ("1 / x" weighting):  $y = 0.148x + 0.00242$  ( $r = 0.9996$ )

